APPENDIX A

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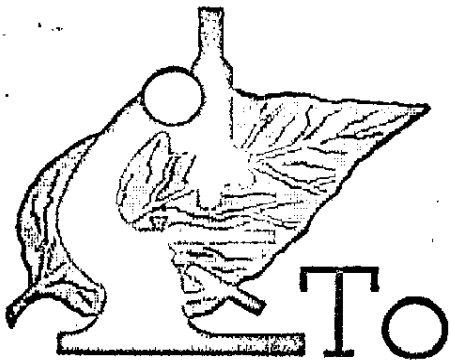

Tobacco Science

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Moisture Content of the Particulate Phase of Smoke from Filter and Nonfilter Cigarettes

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Kingsport, Tennessee, USA

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Moisture Content of the Particulate Phase of Smoke from Filter and Nonfilter Cigarettes

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Introduction

A simple and rapid method for measuring the total particulate matter (T.P.M.) in cigarette smoke is now being used by many laboratories. The method consists of the following: The liquid-solid phase of the smoke from five cigarettes is collected on a tared Cambridge filter assembly, the assembly is reweighed, and from the increase in weight of the Cambridge filter, the total particulate matter delivered by one cigarette may be determined. This method makes no allowance for moisture collected on the Cambridge filter.

Assuming that the moisture content of the T.P.M. from different brands of cigarettes is relatively constant, this method would be satisfactory for comparing the T.P.M. of various brands of cigarettes. However, if the moisture content is not constant, the results could be misleading. Therefore, experiments were carried out to determine whether or not the moisture content of the T.P.M. from different brands of cigarettes varies significantly and to investigate the moisture content of the T.P.M. from filter and nonfilter cigarettes.

Three methods have been reported for measuring the moisture content of the liquid-solid phase of cigarette smoke deposited on an aerosol filter

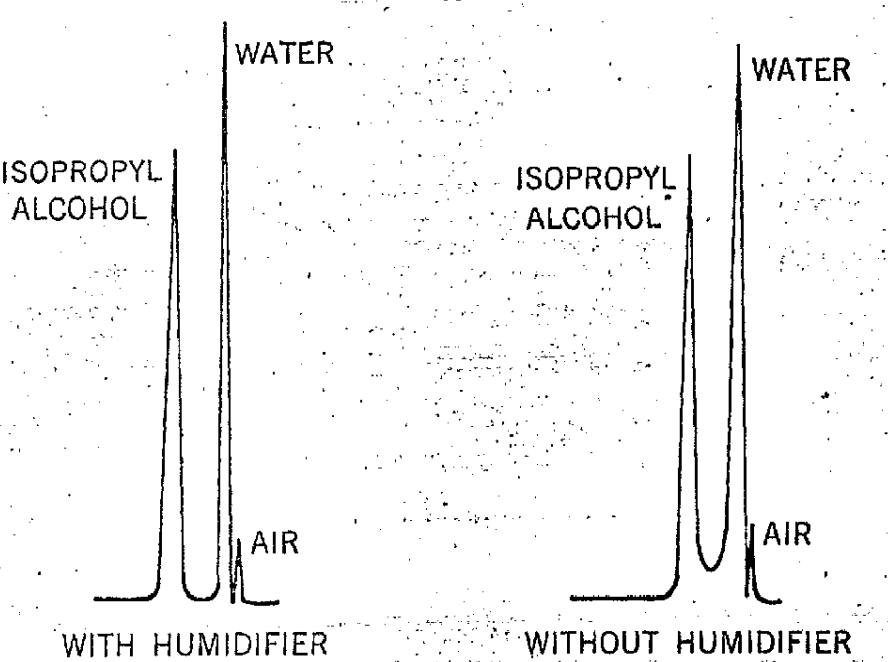

Figure 1. Effect of moisture in carrier gas.

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(Cambridge Number CM-113). The methods are: Karl Fischer analysis as reported by Morgareidge (4) and later modified by Holmes and Cridlin (2), near-infrared spectrophotometric analysis as disclosed by Crowell, *et al.* (1), and gas chromatography as described by Jarrell and Wickham (3). The method reported here is a gas chromatographic method.

Experimental

Reagents. The following reagents were used: p-dioxane (Eastman Kodak Co., redistilled) and isopropyl alcohol (distilled).

Apparatus. The apparatus used comprises the following:

1. Gas liquid chromatography column, ¼ in. × 6 ft., coiled stainless steel tube packed with 40 to 60-mesh Teflon 6 molding powder treated with 10 per cent Dow Silicone oil, DC-550.
2. Leeds and Northrup recording potentiometer.
3. Gow-Mac thermal conductivity cell.
4. Carrier gas humidifier.
5. Hamilton microliter syringe, to deliver 0.1 to 10 μl.
6. Cambridge filter pad (Number CM-113).
7. Lucite Cambridge filter holder.

Operating Conditions. The column temperature was controlled at 70°C and the injection port at 140°C. The helium carrier gas was admitted to the system at a flow rate of 60 ml/min. The carrier gas was conditioned prior to entering the system by a humidifier which was an insulated tube partially filled with 8 ml of water. The added moisture apparently was sufficient to deactivate sites on the column which might absorb polar compounds such as isopropyl alcohol and water. The importance of a humidifier in the system cannot be overemphasized as it minimizes tailing of the water and isopropyl alcohol peaks and makes it possible to obtain reproducible analyses. Figure 1 illustrates the reduced tailing of peaks by use of the humidifier. Figure 2 is a diagram of the gas chromatographic apparatus.

Calibration. The calibration curve shown in Figure 3 was used for converting the chromatographic data into milligrams of water present in the sample. The points on the curve were obtained by analyzing solutions containing known amounts of water in the following manner:

A stock solution of distilled p-dioxane containing 4 ml of distilled isopropyl alcohol per liter was prepared as the water solvent. The isopropyl alcohol was used as an internal standard. The use of an internal standard offers the advantage that the sample size may vary slightly from run to run without affecting reproducibility.

To each of four 20-cc glass vials, 10 ml of the dioxane-isopropyl alcohol solvent was added and each vial was immediately sealed with a serum cap. Water, 5, 10, and 20 ml, respectively, was added to the first three vials. The fourth vial was used as a reagent blank.

The four samples were then analyzed for water by the Karl Fischer method and by gas chromatography. This was accomplished by removing 3-μl samples through the serum caps for the chromatographic analysis and using the remainder of the solution for the Karl Fischer analysis.

In each case the peak heights obtained for the water and the isopropyl alcohol were measured and the peak-height ratio was determined. The peak-height ratios were plotted against milligrams of water found in the samples by the Karl Fischer analysis.

A direct calibration may be made without using the result of the Karl Fischer analysis as a standard if the dioxane is completely dry initially.

Propyl alcohol was also tried as an internal standard instead of isopropyl alcohol. Preliminary studies indicated that it is as good as isopropyl alcohol for this purpose.

Procedure. Five cigarettes, equilibrated to 12 per cent moisture content and weighing within ±20 mg of their average weight, were smoked to a butt length of 30 mm. The smoking system consisted of the cigarette, a tared Cambridge filter assembly, and a smoking machine which produces a 35-ml puff of 2-sec duration at the rate of one puff per minute. The filter assembly was described by Touey (6) and the smoking machine by Mumpower *et al.* (5).

After the last cigarette was smoked, the filter assembly was weighed and opened, and the pad was placed in the sample vial. The interface of the filter assembly was quickly wiped dry with part of a new filter pad, and this was also added to the vial. Dioxane-isopropyl alcohol solution (10 ml) was added, and the vial was sealed with a serum cap and agitated on a mechanical shaker for 20 min.

A 3-μl aliquot of the solution was removed from the sealed vial with a 10-μl syringe and analyzed for water

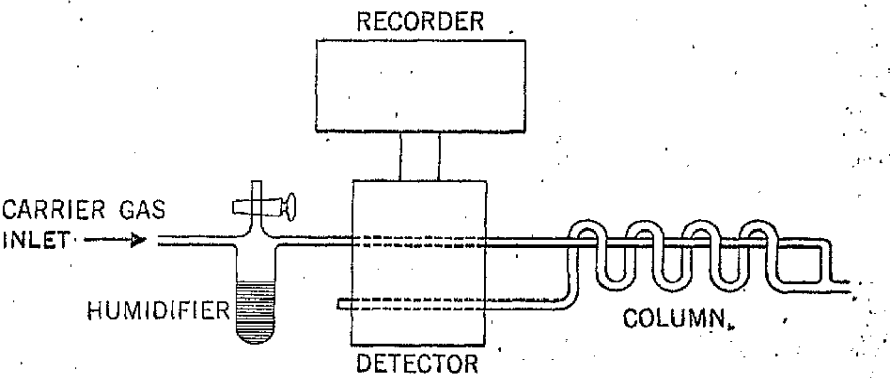

Figure 2. Gas chromatography apparatus.

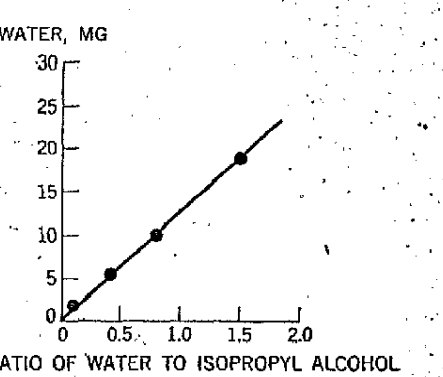

Figure 3. Calibration curve for moisture analysis.

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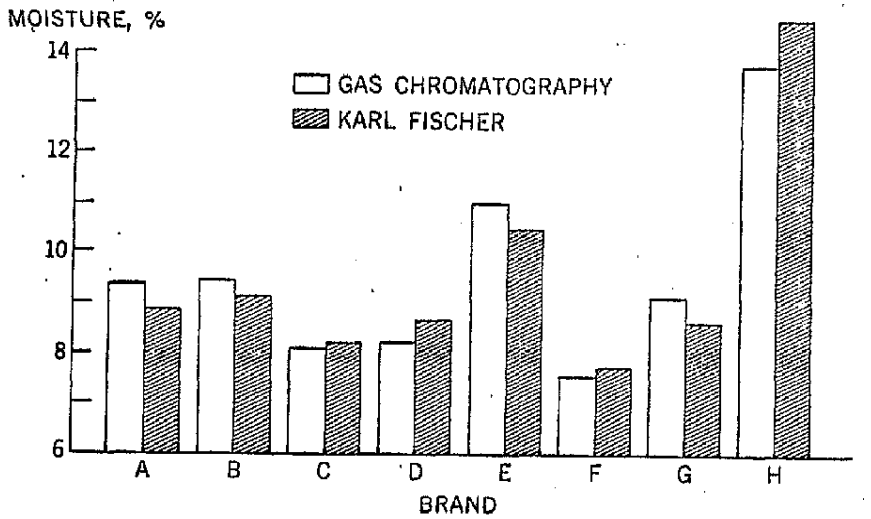

Figure 4. Moisture in T.P.M. of different brands.

content by gas chromatography. The remainder of the solution was analyzed by the Karl Fischer procedure.

In interpreting the gas chromatographic results, the ratio of the peak height of water to the peak height of isopropyl alcohol was obtained. by using this ratio and a calibration curve, the water (in milligrams) in the sample was calculated. From this value was subtracted the value obtained from the analysis of the blank sample.

Results and Discussion

The first experiment was run to compare results of analyses by the Karl Fischer and gas chromatography methods. Eight brands of filter and nonfilter cigarettes were smoked, and each sample of T.P.M. collected was analyzed for moisture by both methods.

A graphic comparison of the results is shown in Figure 4. Agreement between the two methods was generally good. However, the gas chromatographic technique was preferred because the entire sample was not required for the analysis, and there were no reagents which were unstable or difficult to prepare. The method is direct, calibration is simple, and a minimum of technique is required.

The next experiment consisted of smoking the same brand of cigarettes with and without filters and determining the moisture content in the T.P.M. by the gas chromatographic technique (Figure 5). Filters were attached to the 85-mm nonfilter cigarettes and the cigarettes were then shortened to an 85-mm length. As described previously, the cigarettes were equilibrated to a moisture content of 12 per cent and selected on the basis of their average weight. The filters were conditioned at a relative hu-

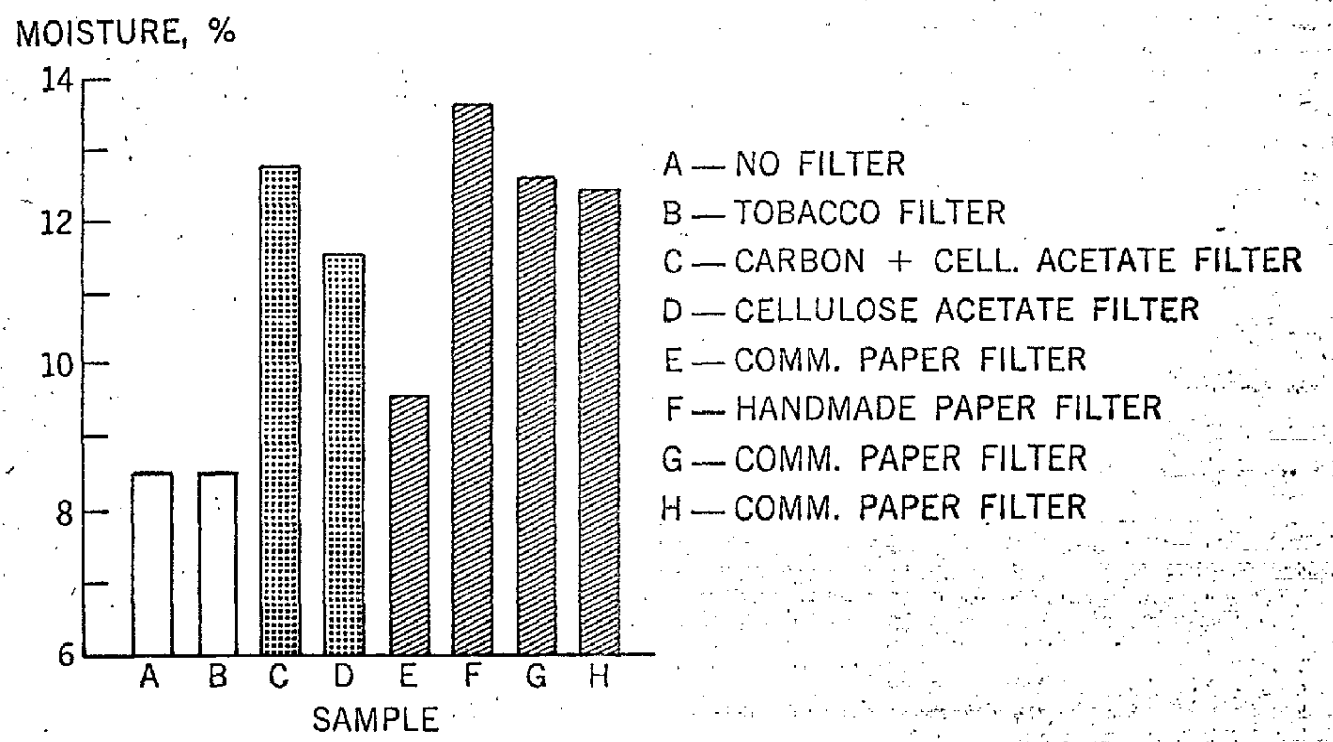

Figure 5. Moisture in T.P.M. of brand K cigarettes with and without filters.

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midity of 66 per cent.

The first sample, A, represented the 85-mm control cigarettes. The next sample, B, was a control cigarette containing a compressed tobacco filter, 17 mm in length, and having a pressure drop of 2.6 in. Sample C had a dual-type filter containing activated carbon with cellulose acetate. Sample D contained a 2-den/fil cellulose acetate filter. The four remaining samples contained paper filters, all of which were commercial filters, except the one in Sample F, which was a handmade paper filter. The values for the moisture content of the T.P.M. obtained from the cigarettes containing cellulose acetate filters were 11.5 per cent and 12.8 per cent. The values for the moisture content of the T.P.M. obtained from cigarettes containing commercial paper filters ranged from 9.5 per cent to 12.6 per cent. The value (8.5 per cent) obtained for the moisture content of the T.P.M. from the control cigarette and from the cigarette which contained a tobacco filter was lower than the values obtained for any of the filtered cigarettes. The value for the moisture content of the T.P.M. from Sample E, which contained a commercial paper filter, was lower than the values obtained for the other paper filters, but was still higher than the value obtained for the control cigarette and the cigarette with a tobacco filter. This higher moisture content for T.P.M. obtained from cigarettes with a cellulose acetate or paper filter was quite unexpected.

The last experiment consisted of determining the moisture content of the T.P.M. obtained from 10 brands of filter and nonfilter cigarettes. Two of the five nonfilter brands were regular size. The five filter brands all contained cellulose acetate filters. The results are shown in Figure 6.

Again it was observed that the value for the moisture content of the T.P.M. from cigarettes with cellulose acetate filters was higher than the value for other filters. The average moisture content for the filter cigarettes was 11.2 per cent, whereas the average for the nonfilter cigarettes was 8.2 per cent.

The standard deviation for the method was calculated from the percentage moisture in the T.P.M. found from 10 individual T.P.M. determinations on control cigarettes. This is shown in Table 1.

The relative standard deviation for the method was 2.3 per cent. The parameters involved included the gas chromatographic separation, smoking of the cigarette, and the variation in moisture content of the T.P.M. for the various groups of control cigarettes used.

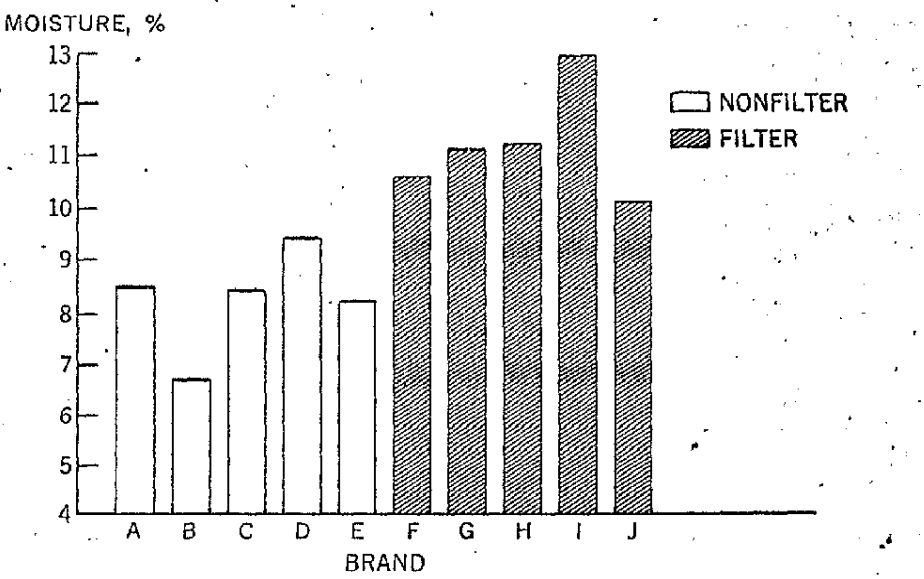

Figure 6. Moisture in T.P.M. from smoke of filter and nonfilter cigarettes.

Summary

The moisture content of the T.P.M. of the smoke from several brands of cigarettes was determined using a gas chromatographic technique. The T.P.M. analyzed by this method consisted of that part of the smoke which was collected on an aerosol (Cambridge) filter pad situated directly behind the cigarette.

The moisture content of the T.P.M. of the smoke from the nonfilter cigarettes tested varied from 6.7 per cent to 9.5 per cent (average value, 8.2 per cent). In the commercial filter cigarettes, the moisture content varied from 10.1 per cent to 12.8 per cent (average value, 11.2 per cent).

The values found for moisture content can be used to calculate the T.P.M. on a dry-weight basis.

When the T.P.M. of different brands of cigarettes or of filter and nonfilter cigarettes is reported, a correction for moisture content makes the comparison much more comprehensive.

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3. Jarrell, J. E. and J. E. Wickham, The determination of moisture in the total particulate matter of cigarette smoke, *Tobacco Sci.*, 6, 154-157 (1962).
4. Morgareidge, F. S., Observations on the determinations of total solids in cigarette smoke, paper presented at the 12th Tobacco Chemists' Conference, Durham, N.C., Oct. 23-24, 1959.

Table 1. Standard deviation for the method.

Moisture in T. P. M., %
8.24
8.23
8.69
8.65
8.66
8.25
8.54
8.49
8.64
8.38
Mean 8.47

Relative standard deviation = 2.3 per cent

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